

Performance Characteristics of the Agilent 7500ce - The ORS Advantage for High Matrix Analysis

Part 1 of a 3 part series on Environmental Analysis

Application

Environmental Analysis

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Abstract

The Agilent 7500ce ICP-MS was specifically designed and optimized for the analysis of trace metals in high matrix samples including environmental, clinical, geological, and others. The 7500ce uses enhanced Octopole Reaction System (ORS) technology for improved sensitivity and robustness over previous generation inductively coupled plasma mass spectrometry (ICP-MS) instruments. This application note outlines the theory of interference removal using the ORS, the design enhancements employed, and the typical performance of the Agilent 7500ce.

Introduction

This application note is Part One of a three part series on environmental analysis using the Agilent 7500ce ICP-MS system. Part Two is an application note demonstrating the ability of the Agilent 7500ce ICP-MS system to measure trace elements in drinking water, at substantially below regulated levels, under challenging real-world conditions [1].

Part Three is an application note covering the analysis of various high matrix environmental samples using the Agilent 7500ce ICP-MS [2].

This application note details the advances in ion optics and octopole reaction system (ORS) design that were incorporated into the 7500ce. These advances came about as a result of extensive testing and development of its predecessor (Agilent 7500c) with difficult, high-matrix samples. The design goals of the 7500ce were:

- Develop an ICP-MS system specifically to meet the needs of analytical laboratories to analyze unknown, variable, high-matrix samples, which are currently depending on inductively coupled plasma-optical emission spectroscopy (ICP-OES), graphite furnace atomic absorption (GFAA), and hydride and cold vapor techniques, in addition to ICP-MS.
- Maintain the simple, effective interference removal characteristics of the ORS - successfully introduced in the 7500c.
- Improve the overall sensitivity to allow ultra-trace analysis of mercury (Hg) and other low level elements, which were previously difficult in some very high matrix sample types.

These goals were achieved through enhancements in the sample introduction system, interface, ion optic, and ORS regions of the instrument. In common with all the other models in the 7500 Series, the 7500ce uses highly efficient 27 MHz plasma coupled to a low-flow nebulizer and cooled-spray chamber to minimize plasma and interface matrix effects. This approach has been



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successfully used in all Agilent ICP-MS instruments since the 4500 Series in 1994, but recent enhancements with the development of a new digitally driven, all solid-state RF generator have further increased plasma robustness. This serves to reduce metal oxide interferences, as evidenced by a very low CeO^+/Ce^+ ratio of <1.5% (<0.8% in He cell gas mode). Following the successful strategy of the 7500c, all ion lenses with the exception of the octopole are outside the high vacuum region and can be serviced without venting the mass analyzer. This design greatly reduces downtime for routine system maintenance. The 7500ce maintains a linear, axial flow of ions from the sampler and skimmer cones through a pair of on-axis extraction lenses, enhancing ion transmission and reducing the effects of matrix accumulation on the extraction lenses. Borrowed from the successful 4500 and 7500a systems, the 7500ce uses a simplified Omega lens to eliminate photons and neutrals from the ion beam before entering the octopole. Unlike older photon stop designs, the Omega lens eliminates photons and neutrals while maintaining high ion transmission, particularly at low masses. After the Omega lens, ions enter the octopole

reaction cell, which is now located on-axis to the quadrupole and detector, further enhancing ion transmission. The redesigned ion lens and ORS provide improved ion transmission without compromising the tight control of ion energy, which is essential for efficient interference removal by energy discrimination (ED).

Figure 1 compares the 7500c and 7500ce configurations, highlighting the simplification in the ion trajectory that has led to the improved performance specifications of the 7500ce. Enhancements in software designed specifically for routine high matrix analyses add additional capability and ease of use. These include the introduction of “Virtual Internal Standardization” (VIS) which allows the user to interpolate between internal standard (ISTD) response factors to create a VIS at a mass where no appropriate ISTD exists. Intelligent calibration resloping can automatically fine-tune a calibration curve, if needed, during a long sequence of high matrix samples, without the time consuming recalibration. This can be accomplished in the same process as monitoring a required continuing calibration verification (CCV).

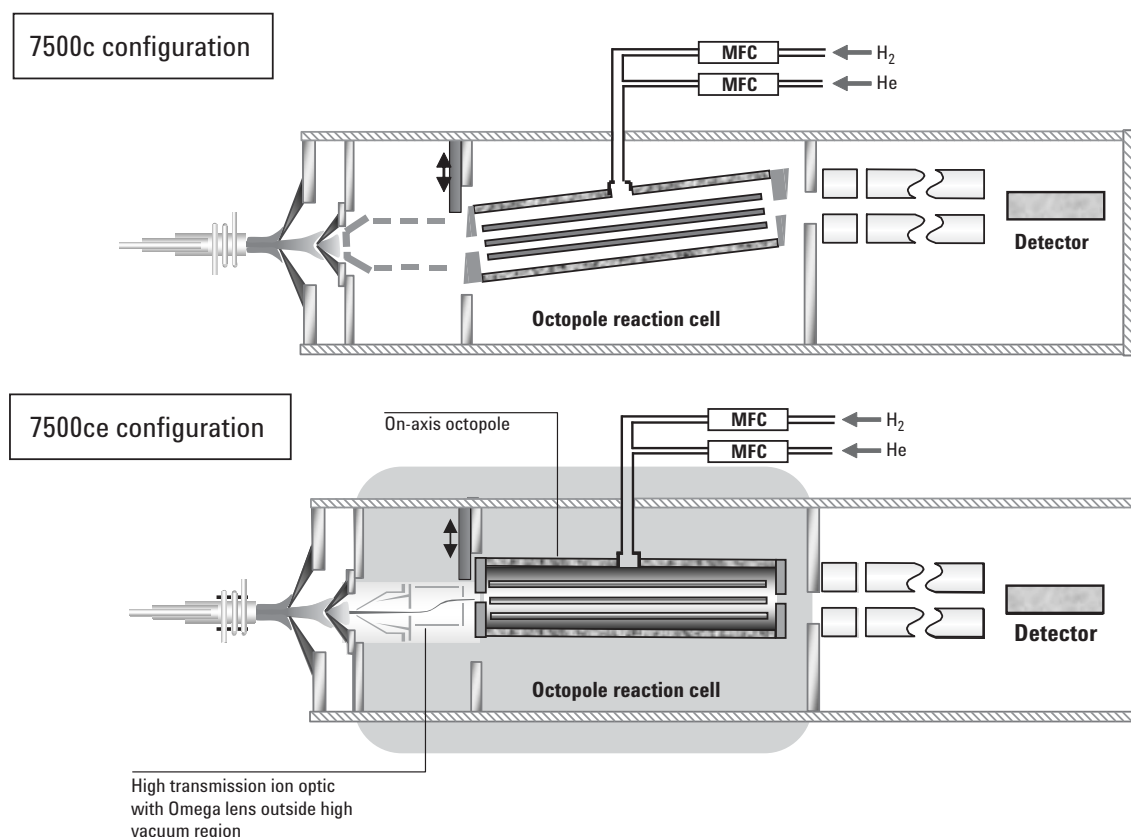


Figure 1. A comparison of the ion optic and octopole configurations between the Agilent 7500c and 7500ce ICP-MS systems.

Enhanced ORS

Like its predecessor the 7500c, the 7500ce uses collision/reaction cell (CRC) technology in the form of the ORS to remove polyatomic interferences. The use of CRC technology to reduce interferences in ICP-MS is well-documented [3]. However, until now, there have been compromises associated with the use of some designs of CRC ICP-MS for trace level, multi-element analysis in unknown or variable matrices. These compromises included poor sensitivity for low-mass analytes, poor stability, and the necessity for matrix matching of samples and standards to avoid unexpected new interferences caused by complex, sequential reaction chemistry in the cell. As a result, some CRC systems allow only the analysis of a small number of analytes under a specific set of conditions for a single sample matrix.

Numerous publications [3, 4, 5] have discussed the mechanisms for polyatomic interference removal using CRC technology including:

- Collisionally induced dissociation (CID)
- Chemical reaction
 - Charge transfer
 - Atom transfer
- Kinetic energy discrimination (KED)

Mechanism 1, CID, does not occur to any great degree with the relatively light gases typically used in the collision cell because the combined kinetic energy of the collision does not generally exceed the bond energy of the polyatomic species. In most CRC ICP-MS systems, chemical reaction mechanisms including charge transfer and atom transfer

are the predominant mechanisms [4, 5]. However, in order to provide sufficient reduction of interferences, the reaction must be highly favored, which can require the use of very reactive gases for many interferences. Such gases can also react with analyte ions, so reducing sensitivity and compromising multi-element analysis, or form reaction by-products that can interfere with other analytes [4]. In this case, reaction cell conditions must be matched to a specific analyte/matrix combination and cannot be used simultaneously for multiple analytes in variable matrices. Mechanism 3, KED, relies on the fact that at the exit of the collision cell polyatomic species will possess lower kinetic energy than atomic ions at the same mass-to-charge ratio [3, 4]. This is due to the fact that collision cross sections of polyatomic ions are larger than for atomic ions, so that polyatomic species suffer more collisions with the cell gas, thus losing more of their initial energy. A bias voltage at the cell exit is then used to reject the low-energy polyatomic species, while allowing the high-energy atomic ions to enter the quadrupole for analysis and detection

Three Modes of Operation - One Set of Conditions

Table 1 lists the typical instrument conditions used for high-matrix analysis for the 7500ce. Instrument parameters are essentially the same for all three modes of operation¹. This is because no complex procedures are required to remove newly created interferences or to avoid the reactive loss of analyte in any ORS mode.

¹Slightly higher bias voltages are required in the octopole and quadrupole to maintain ion velocity in a pressurized collision cell compared with a nonpressurized cell. Other parameters, with the exception of the cell gas flow, are identical in all modes of operation.

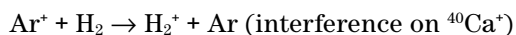
Table 1. Instrument Parameters for Robust Plasma Conditions Used with the 7500ce

Instrument parameter	Normal mode	Hydrogen mode	Helium mode
RF Power	1500 W	<Same	<Same
Sample depth	8 mm	<Same	<Same
Carrier gas	0.85 L/min	<Same	<Same
Makeup gas	0.2 L/min	<Same	<Same
Spray chamber temp	2 °C	<Same	<Same
Extract 1	0 V	<Same	<Same
Extract 2	-160 V	<Same	<Same
Omega bias	-24 V	<Same	<Same
Omega lens	-0.6 V	<Same	<Same
Cell entrance	-30 V	<Same	<Same
QP focus	3 V	-11 V	<Same as H ₂
Cell exit	-30 V	-44 V	<Same as H ₂
Octopole bias	-7 V	-18 V	<Same as H ₂
QP bias	-3.5 V	-14.5 V	<Same as H ₂
Cell gas flow	0	3.0 mL/min H ₂	4.5 mL/min He

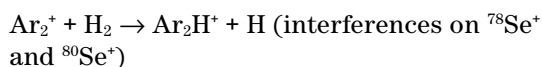
The Hydrogen Reaction Mode

In hydrogen reaction mode, the ORS is pressurized using a small flow of pure hydrogen at 1–5 mL min⁻¹. Simple reactions with hydrogen remove argon-based polyatomics according to the following examples.

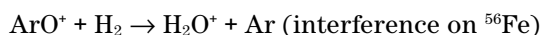
Charge (e⁻) transfer:



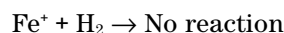
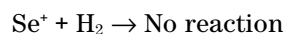
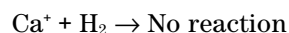
Proton transfer:



Atom transfer:



In all cases the Ar interference is removed from mass 40, 56, 78, and 80. Since Ca, Fe, and Se do not react with H₂, there is no loss of analyte signal.



Note that some of these reaction processes lead to new polyatomic ion species, principally hydrides of the original interference. However, these new, cell-formed species all have low energy and are removed from the ion beam using the same bias voltage, as was discussed above, under interference removal by KED.

Reaction mechanisms can be highly efficient, as evidenced by the calibration curves in Figure 2 for ⁷⁸Se, ⁴⁰Ca and ⁵⁶Fe, which show that all the “normal” background species are reduced significantly under a single set of cell conditions.

Figure 3 illustrates the reduction in background from Ar⁺ at *m/z* = 40 as hydrogen flow in the cell is increased, yielding a 10⁹ reduction in background. Since the reaction chemistry is specific to argon polyatomics, no signal is lost due to reaction of the analyte with hydrogen, as could occur with other more reactive gases. However, due to the specificity of reaction mode, there are numerous examples where it is not useful. For example, in samples where the matrix composition is unknown, or there are multiple polyatomic interferences at a single *m/z*, it is not possible to use reaction mode effectively. In this case, a more generic method of interference removal is needed.

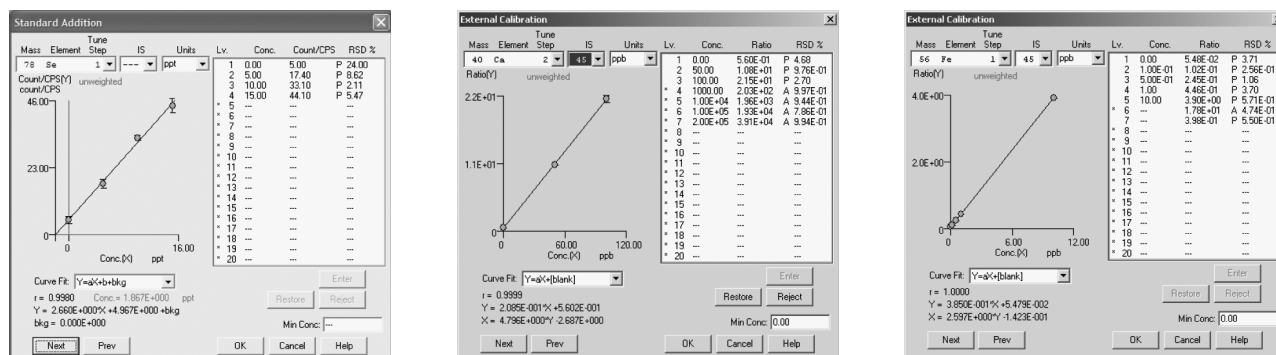


Figure 2. Calibration plots for ⁷⁸Se, ⁴⁰Ca, and ⁵⁶Fe under hydrogen reaction conditions.

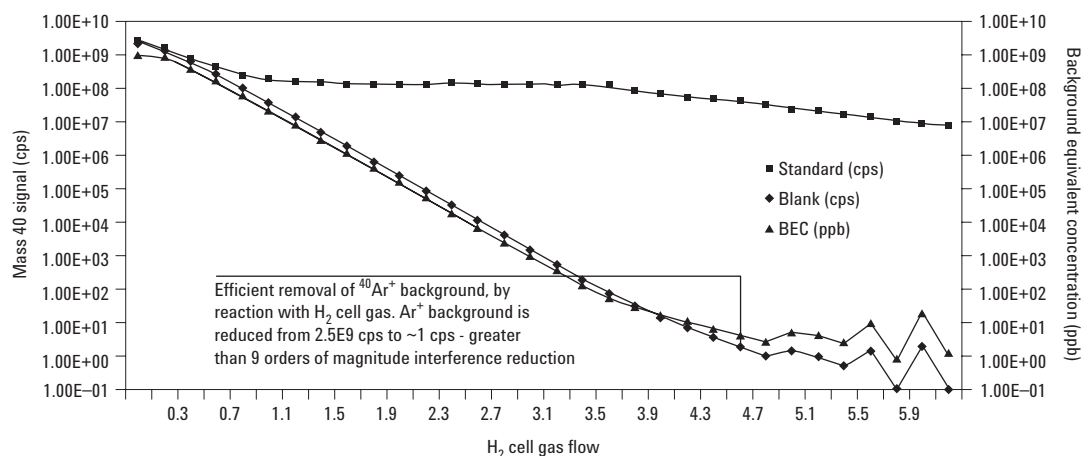


Figure 3. Reduction in background on mass 40 for calcium using hydrogen reaction mode. In this case, as the hydrogen flow is increased to about 5 mL/min, the background at mass 40 decreased from approximately 2.5 billion cps to about 1 cps, a $>10^9$ reduction.

The Helium Collision Mode

Helium collision mode can reduce or eliminate polyatomic interferences by one of two mechanisms; either CID or KED. Both are nonreactive mechanisms and so they do not form any new polyatomic ionic species that must be managed. CID can occur when the collision energy between the polyatomic ion and the collision gas (typically He) is sufficient to break the polyatomic bond. The result is two (usually atomic) fragments at lower mass, one of which will retain the charge of the original ion. A few common polyatomic

interferences are bound weakly enough for this to occur. They include NaAr^+ , which can interfere with the measurement of ^{63}Cu in high sodium samples and ArO^+ which interferes with iron. However, when ion energies are properly controlled, KED is the more useful of the two techniques. Kinetic energy discrimination depends on the fact that polyatomic ions are always larger in collisional cross section than monatomic ions (Figure 4), and as a result undergo more collisions and so lose more energy when traversing a pressurized collision cell. Figures 5 and 6 depict the KED process.

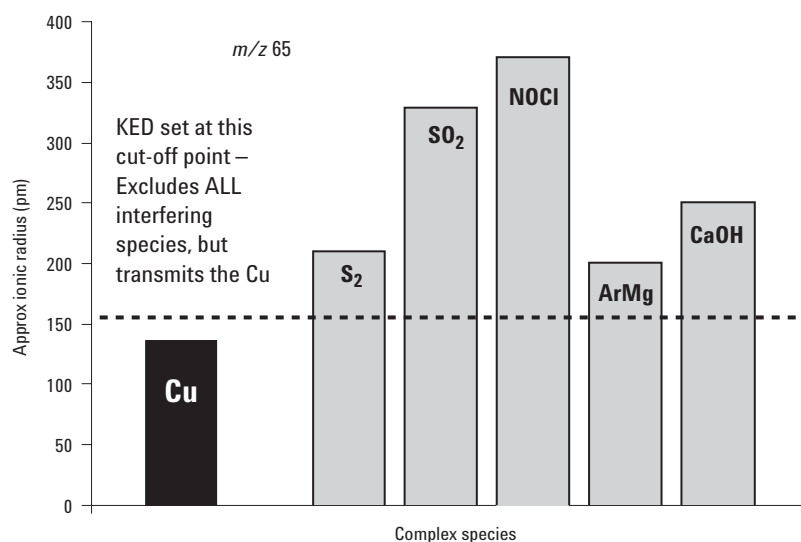


Figure 4. Comparison of approximate ionic radii (in picometers) for copper and several polyatomic species.

Figure 5 shows the greater loss of energy of the polyatomic ion relative to the atomic ion, in this case ArCl^+ relative to As^+ . However, for KED to be effective, it must remove the polyatomic ion effectively while not significantly reducing the response of the atomic ion. This means there must be minimal overlap in ion kinetic energies between the polyatomic and atomic ions at the exit of the octopole. For this to be the case, the energy spread of incoming ions must be less than the energy difference between analyte and polyatomic interference at the octopole exit.

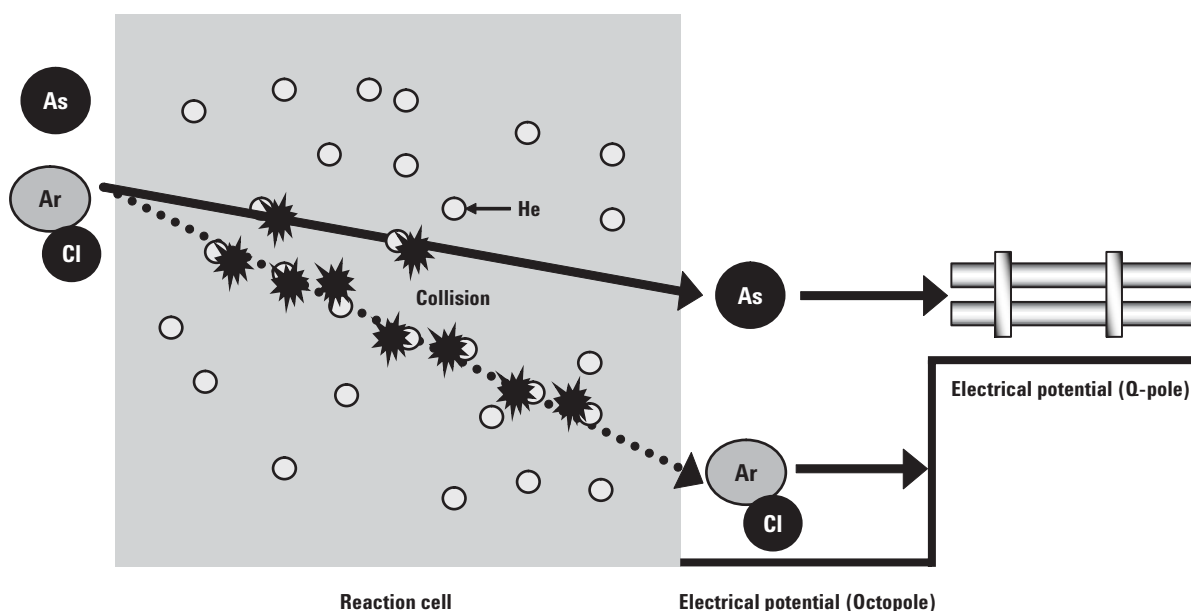


Figure 5. KED. Polyatomic species have a larger collision cross section, and so experience more energy dissipating collisions and exit the cell with lower kinetic energy. A small stopping potential between the exit of the octopole and the entrance of the quadrupole keeps the polyatomic ions from entering the quadrupole and being detected.

Only the Agilent ORS can accomplish this, as a result of the use of the ShieldTorch system, which minimizes plasma potential and eliminates secondary ionization in the interface, which would otherwise cause broadening of the ion energy spread. It is also essential to avoid band-broadening collisions induced by high extraction voltages in the high-pressure region immediately behind the skimmer cone. On the 7500ce, this is accomplished by using soft-extraction, (extract 1 operates at 0 to +5 V), as a result of which, the mean ion energy is maintained at less than 2 eV with an ion energy spread of about 0.5 eV, ideal for the KED of plasma-source polyatomic interferences.

A simplified schematic representation of ion kinetic energy and energy distribution for a typical ICP-MS system and from an Agilent 7500 ORS ICP-MS is shown in Figure 6.

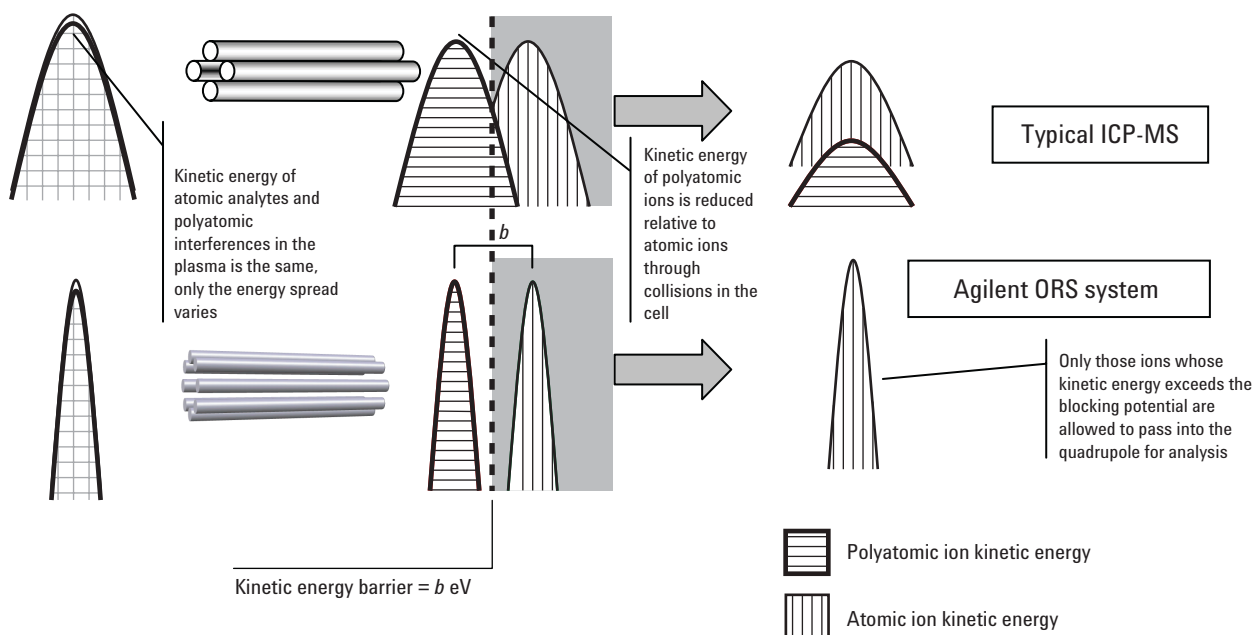


Figure 6. Simplified schematic representation of ion kinetic energy and energy distribution of a typical ICP-MS system (upper), and from an Agilent 7500 ORS ICP-MS (lower).

After multiple collisions in the CRC, in both cases the average kinetic energy of the larger polyatomic ions is decreased relative to the smaller atomic ions by b eV. If a kinetic energy barrier is applied to the ion beam at the exit to the collision cell that is equivalent to the kinetic energy difference, b , (indicated by dashed line), then any ions whose kinetic energy is lower than the barrier will be blocked. If the energy distribution of ions is larger than the difference in average energies of the ions, only partial rejection of polyatomic ions occurs accompanied by a loss of atomic ions.

While ED was described on other designs of ICP-MS systems, these systems do not have the tight control of ion energy provided by the ShieldTorch System and so the ED is only effective for reducing the very low energy polyatomics formed within the cell, typically as a result of sequential reaction chemistry, which is characteristic of the use of a highly reactive cell gas, such as NH_3 . Table 2 shows the reactants and products for a number of polyatomic interferences using both hydrogen and ammonia reaction mode. As can be seen, in the presence of common matrix components such as carbon and sulfur, the use of NH_3 can create multiple new interferences, which must be removed. Avoiding the use of NH_3 eliminates the possibility of creating new, cell-formed, polyatomic cluster ions in the first place.

Reaction of hydrogen with plasma-based polyatomics such as Ar^+ is highly favored and results in elimination of the interferent. Neither hydrogen nor NH_3 are effective at removing the interference from ClO^+ on vanadium. Additionally, the use of ammonia can lead to reaction with other common matrix elements such as carbon and sulfur, creating new interferences such as HCN^+ on aluminum and NHSH^+ on titanium. Using the Agilent system with an inert cell gas and KED would eliminate the interference from ClO^+ on vanadium AND, for example, ArC^+ on Cr, without producing any new interferences.

An excellent test of the efficiency of interference removal can be seen in low-level calibration plots. When interferences are present, the response curve will be offset in the y direction by the magnitude of the interference, increasing the background equivalent concentration (BEC) and the detection limit (DL). When the interference is removed, the calibration curve intersects the y-axis at a point much nearer to zero with a correspondingly lower BEC and DL. Figure 7 depicts sub-ppb calibration curves for chromium and vanadium in 1% each methanol, HCl, and HNO_3 , with and without the use of helium collision mode. Since KED does not depend on chemical reaction, it is independent of matrix concentration as well as composition.

Table 2. Comparison of Reaction Products for Several Possible Reactants Involving Hydrogen and Ammonia

Reactants		Interfered analytes	Products	
Ar^+	H_2	Ca^+	H_2^+	Ar
Ar_2^+	H_2	Se^+	ArH^+	Ar, H
ArO^+	H_2	Fe^+	H_2O^+ , ArOH^+	
ClO^+	H_2	V^+	None	None
ClO^+	NH_3	V^+	None	None
HCN^+	H_2	Al^+	HCNH^+ (28)	H
C^+	NH_3		NH_3^+ (17)	C
			HCN^+ (27)	H_2
			HCNH^+ (28)	H
S^+	NH_3		NHSH^+ (48)	

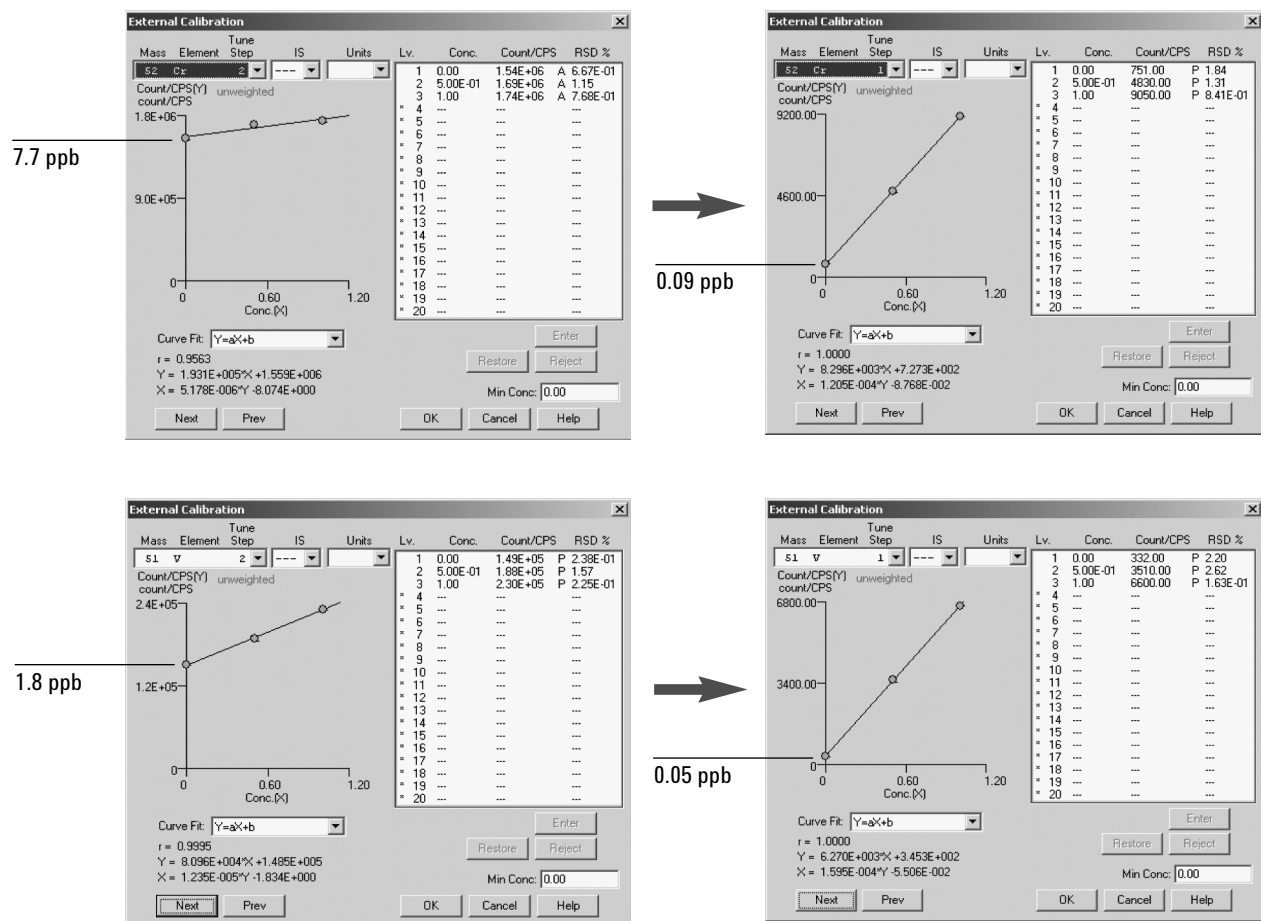


Figure 7. Calibration plots of ^{52}Cr and ^{51}V in 1% nitric, 1% hydrochloric, 1% methanol showing the contribution of interferences from ArC^+ and ClO^+ in normal mode on the left and after removal of interferences by the ORS using He on the right.

Figure 8 depicts the effect of increasing HCl on the measured concentration of a 5-ppb solution of vanadium in both helium and normal (no gas) mode. Increasing the HCl from 0% to 1% causes an 80% increase in measured vanadium concentration in the no-gas mode. There is no increase in the V concentration reported for the variable sample matrix, when measured in He mode.

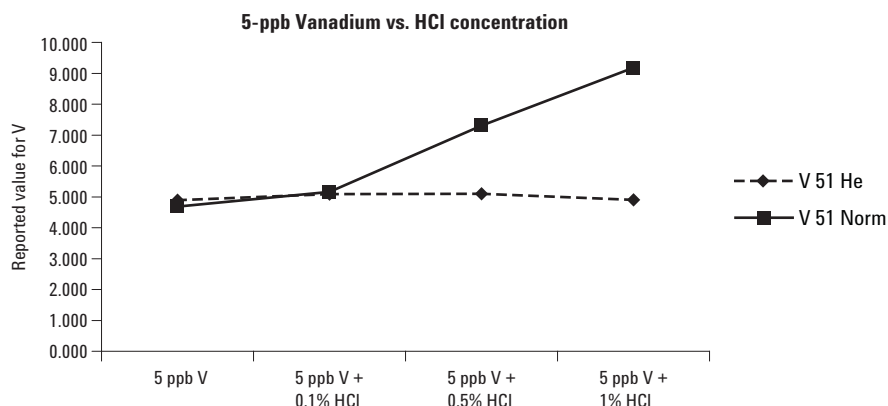


Figure 8. Effects of increasing HCl concentration on vanadium response in both normal and helium modes.

The Normal Mode

Normal mode uses the octopole as an ion guide only with no additional gas added. In this mode, the Agilent 7500ce ICP-MS operates as a conventional (non-collision cell) instrument. Because an octopole is a highly efficient ion guide as compared with a lower-order multipole, such as a hexapole or quadrupole, there are no compromises in ion transmission efficiency and the addition of a collision gas to promote collisional focusing in normal mode is not required. Because of this, the Agilent

7500ce exhibits exceptional sensitivity for uninterfered low mass elements, such as lithium, beryllium, and boron. Typically, normal mode is used only for these elements, though it is also acceptable for other elements that do not require interference removal such as lead, mercury, thallium, and uranium. Examination of Table 3 will show that the DLs for the interference-free heavy metals are essentially the same in all three modes, giving the user the flexibility to select as appropriate.

Table 3. Three Sigma Detection Limits (ppt). All Isotopes 1 s Total Integration Time Except Ca and Hg Which Were 3 s Total Integration Time

Mass	Element	STD Mode (No gas)		H ₂ Mode (5 mL/min)		He Mode (4 mL/min)	
		DL (3-Sigma)	BEC	DL (3-Sigma)	BEC	DL (3-Sigma)	BEC
6	Li	4.99	17.09	161.491	95.83	331.32	142.58
7	Li	1.67	14.36	23.755	26.31	9.38	20.55
9	Be	0.19	0.11	6.932	2.62	7.812	2.61
11	B	5.88	47.26	83.182	128.03	48.21	107.28
23	Na	3.36	148.40	62.682	313.64	37.65	299.38
24	Mg	0.27	0.72	2.570	1.75	3.37	1.41
27	Al	3.05	50.70	8.079	5.51	37.56	53.52
31	P	418.27	12521.95	—	—	1903.62	3800.52
39	K	1347.50	47564.25	29.532	118.74	2838.73	27943.17
40	Ca	—	—	2.936	7.13	—	—
43	Ca	460.04	8520.74	129.640	121.68	191.85	742.92
44	Ca	2932.48	50407.443	102.104	121.94	48.01	352.38
45	Sc	7.95	183.06	6.446	19.41	1.34	6.46
47	Ti	4.07	40.86	32.197	19.88	4.98	3.52
49	Ti	11.49	57.33	17.535	10.46	5.69	1.70
51	V	0.40	2.52	1.309	0.73	0.42	0.19
52	Cr	5.53	212.670	19.919	68.63	3.10	22.70
53	Cr	7.98	52.87	28.504	82.65	8.70	21.60
55	Mn	1.69	25.24	1.362	4.10	4.25	12.23
56	Fe	1443.70	55093.34	5.034	20.21	53.99	451.26
57	Fe	444.36	23132.21	66.614	261.56	71.06	215.90
59	Co	0.21	2.15	0.816	0.26	0.38	0.30
60	Ni	26.523	672.20	71.224	742.27	41.70	491.67
63	Cu	1.32	37.48	20.271	20.14	6.37	68.35
65	Cu	2.70	47.46	27.845	27.76	7.52	59.76
66	Zn	2.85	9.01	1.612	1.83	1.84	2.18
69	Ga	0.30	3.03	0.273	0.19	0.82	1.04
71	Ga	1.22	7.40	0.125	0.15	1.80	2.22
72	Ge	2.60	53.69	1.448	1.14	3.32	7.55
73	Ge	3.72	32.74	5.556	2.74	7.30	6.04
75	As	23.24	660.78	14.130	14.00	10.72	65.72
78	Se	48.10	6351.29	2.396	2.556	48.93	195.45
82	Se	26.92	251.29	15.225	56.51	20.63	116.01
85	Rb	0.27	1.06	0.349	0.57	0.72	0.34
88	Sr	0.19	0.84	0.072	0.04	0.38	0.13
89	Y	0.10	0.26	0.054	0.04	0.16	0.04
90	Zr	0.07	0.09	0.709	0.18	0.15	0.06

Table 3. Three Sigma Detection Limits (ppt). All Isotopes 1 s Total Integration Time, Except Ca and Hg Which Were 3 s Total Integration Time (Continued)

Mass	Element	STD Mode (No gas)		H ₂ Mode (5 mL/min)		He Mode (4 mL/min)	
		DL (3-Sigma)	BEC	DL (3-Sigma)	BEC	DL (3-Sigma)	BEC
93	Nb	0.12	0.20	0.68	0.53	0.15	0.05
95	Mo	0.32	0.67	14.53	4.33	0.31	0.1
101	Ru	0.60	1.40	19.27	5.74	0.28	0.09
103	Rh	0.08	0.11	4.24	1.75	0.05	0.0
105	Pd	0.30	0.27	10.11	6.39	0.33	0.15
107	Ag	0.23	0.33	1.42	1.61	0.28	0.4
111	Cd	0.56	0.83	0.32	0.20	0.86	0.54
115	In	0.07	0.11	0.05	0.03	0.08	0.05
116	Cd	0.33	0.41	0.40	0.47	0.34	0.23
118	Sn	0.24	0.43	0.55	0.54	0.91	0.73
121	Sb	0.11	0.08	0.21	0.010	0.46	0.25
125	Te	1.96	0.94	2.05	1.29	9.57	4.12
126	Te	1.12	1.64	2.08	1.67	7.33	4.27
127	I	2.02	21.73	3.57	22.30	7.71	20.41
133	Cs	0.09	0.04	0.06	0.04	0.15	0.06
137	Ba	0.22	0.20	0.38	0.16	0.9	0.38
139	La	0.17	1.94	2.49	2.46	0.66	2.14
140	Ce	0.223	2.65	2.18	3.21	0.47	2.88
141	Pr	0.11	0.25	0.12	0.28	0.12	0.31
146	Nd	0.39	0.44	0.43	0.50	0.70	0.73
147	Sm	0.22	0.17	0.11	0.04	0.58	0.24
153	Eu	0.02	0.03	0.04	0.02	0.11	0.03
157	Gd	0.17	0.14	0.15	0.05	0.35	0.22
159	Tb	0.03	0.02	0.01	0.01	0.055	0.03
161	Dy	0.18	0.15	0.05	0.05	0.23	0.17
163	Dy	0.15	0.08	0.08	0.04	0.23	0.16
165	Ho	0.04	0.00	0.02	0.01	0.05	0.02
166	Er	0.15	0.06	0.05	0.02	0.17	0.09
169	Tm	0.02	0.02	0.02	0.01	0.03	0.03
172	Yb	0.11	0.09	0.07	0.02	0.30	0.18
175	Lu	0.04	0.020	0.02	0.01	0.06	0.03
178	Hf	0.13	0.08	0.06	0.06	0.32	0.15
181	Ta	0.04	0.06	0.06	0.049	0.11	0.08
182	W	0.32	0.35	1.39	0.5	0.56	0.33
183	W	5.07	1.07	0.87	0.42	0.43	0.47
185	Re	0.12	0.07	0.07	0.05	0.12	0.08
193	Ir	0.09	0.10	0.25	0.08	0.33	0.15
195	Pt	0.14	0.17	1.94	0.52	0.22	0.18
197	Au	0.22	0.11	1.76	0.43	0.18	0.07
200	Hg	0.82	2.00	1.04	1.78	1.15	2.18
201	Hg	1.11	2.54	2.07	2.29	1.56	2.58
202	Hg	0.86	1.84	0.75	1.77	0.59	1.91
205	Tl	0.20	0.24	0.13	0.22	0.35	0.30
206	Pb	0.33	0.84	0.28	0.64	0.34	0.73
207	Pb	0.51	0.94	0.25	0.69	0.95	1.05
208	Pb	0.47	0.712	0.40	0.55	0.53	0.755
209	Bi	0.05	0.04	0.03	0.02	0.06	0.05
232	Th	0.04	0.04	0.03	0.01	0.06	0.05
238	U	0.05	0.04	0.04	0.01	0.05	0.044

Conclusions

The Agilent 7500ce ICP-MS has achieved its design goals of providing sensitive, robust, interference-free analysis of difficult, high-matrix samples. With five times the sensitivity of its predecessor, nine operating orders of dynamic range and increased matrix tolerance, it is capable of replacing both GFAA and ICP-OES instruments in addition to older generation ICP-MS systems. The 7500ce is unique in offering a single solution for multi-elemental analysis of complex and variable, high matrix samples, while allowing the operator the freedom to use simple and consistent sets of instrument conditions for almost all elements in almost any matrix.

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